VOL. 11 NO. 1,
FEBRUARY, 2026
E-ISSN 3027-0995
P-ISSN 3026-8265

African Scholar Publications & Research International
www.africanscholarpub.com

Journal of Biotechnology and Agricultural Research (JBAR)

Assessment of Pesticide Residues in Soil and Cassava using GC-MS in Obiaruku, Delta State, Nigeria

Thompson Aiyevbekpen Ehigiegba; Osadebe Biose; Blessing Obatuebise Olatunji; Akinjagunla Akinmoladun; & Effiong Eyo Effiong

National Centre for Energy and Environment, Energy Commission of Nigeria, University of Benin, Benin City, Nigeria.
Corresponding Author: ehitomtom@gmail.com
DOI: https://doi.org/10.70382/ajbar.v11i1.029

Abstract

The problem of pesticide residues in soil and crops, such as cassava, is becoming increasingly a concern because it may have adverse effects on environmental health and food safety. This research examines the pesticide residues in the cassava tubers and from farms in Obiaruku, Ukwuani LGA, Delta State, Nigeria to determine the level of contamination and hazard. The purpose was to determine the presence and level of pesticide residues in soil and cassava and the probable effects it has on the health and environment of the people. Systematic grid sampling was used to sample soil and cassava samples. Soxhlet and ultrasonication were used to extract pesticide residues, which were determined by using Gas Chromatography-Mass Spectrometry (GC-MS). Principal Component Analysis (PCA) and Factor Analysis were utilised to determine the distribution and correlation of pesticide residues. The findings indicated that the cassava roots and soil were seriously polluted with pesticides like 1, 3-dimethyl- 2- nitro benzene, Fenitrothion, Pirimiphos Ethyl and Phosalone in different amounts at different sites of investigations. Pesticide concentration in cassava tubers was lower than in the soil, indicating that there was not much translocation to the edible portion. The Cassava tuber samples had lower pesticide concentrations compared to soil, with the highest pesticide concentrations in soil being Fenitrothion at 45.99 ± 76.62 mg/kg and Pirimiphos-ethyl at 38.32 ± 52.91 mg/kg. The cassava residue samples contained very low pesticide concentrations, with the highest concentration in the residue sample being Pirimiphos-ethyl at 1.024 ± 1.65 mg/kg. Both Fenitrothion and Pirimiphos-ethyl were found to be some of the most predominant soil

pollutants, although their concentrations were significantly lower in cassava ($p<0.05$ Nevertheless, the prevalence of pesticides in soil presents a major concern to the well-being of the environment and food safety. Therefore, the frequent observation of pesticide residues, adoption of eco-friendly farming methods, and enhanced pest control strategies are advised to avoid pollution. Moreover, increased awareness of the risks of pesticides and the availability of safer variants should be considered a priority issue.

Keywords: GC-MS, Principal Component Analysis, Food Safety, Organophosphate Pesticides, Delta.

Introduction

Agricultural productivity is an important factor in food security, economic growth, and sustainable development of most developing nations (Cahyaningsih and Rahayu, 2023). Sub-Saharan African countries and Nigeria especially, use cassava (*Manihot esculenta*) as a staple crop growing on their soils that provides carbohydrates to millions of Africans. The reason for cultivation is its ability to grow on poor soils and in dry weather, as well as the variety of its applications in the human diet and industry (Adebayo et al., 2020; Adebayo, 2023). Nonetheless, crop production and yield have been on the rise with an increasing demand of pest-free produce and crops, prompting the widespread use of pesticides in production (Mwebaze et al., 2022). Although pesticides have been useful in combating pests, diseases, and weeds, careless use is harmful to the environment and human health, particularly when traces are left in food crops and the soils around them (Osuji et al., 2024). By definition, pesticides are biologically active compounds that are designed to control or kill pests. Nonetheless, many of these chemicals have long half-lives, and they can bioaccumulate in the ecosystems (Schlieffer and Speiser, 2022). When overused or misused, they may seep into the soil and get taken up by root crops, which then find their way into the food chain. This casts significant doubts on food safety, especially in root and tuber crops such as cassava, with roots in soil and direct contact to possibly contaminated soils (Gomes et al., 2020). The persistent nature of some pesticide residues means that even after harvest, traces of these chemicals can remain in cassava tubers, posing a potential health hazard to consumers through chronic exposure (Ali et al., 2021)

Moreover, the environmental consequences of pesticide residues in soil are drastic. Pesticides in soils can destroy the microbes in soils, decrease soil fertility, and modify nutrient cycles (Sharma et al., 2019). This damages the health of the soil, which jeopardises the long-term production and sustainability of agriculture. Moreover, residues can also cause pollution of the environment by leaching and runoff into groundwater and surrounding water bodies (Ali et al., 2021; Kumar et al., 2021). The lack of awareness regarding the handling of pesticides in areas where subsistence and

small holder farming are the most frequent, the absence of regulation enforcement, and poor agricultural extension services are factors that lead to unsafe pesticide application which allows for soil and crop contamination (Gomes et al., 2020).

Contamination of cassava with traces of pesticides may result in food-borne diseases and long-term health complications such as hormonal imbalance, neurological diseases and even cancerous conditions (Ali et al., 2021). Populations at risk include vulnerable groups like children, expectant women, and immunocompromised people. Even with these risks, pesticide residue in food crop and soil data in most of Africa are scanty (Kumar et al., 2021). Local investigations to assess the level of contamination and detailed information on the compounds of pesticides used, and the possible health and ecological risks are urgently required (Zhao et al., 2020).

Studies have been done to study pesticide residues in leafy vegetables and grains, but there is an observed gap in the literature on root crops such as cassava. As the roots are always in touch with the soil then it is likely to accumulate more residues than aerial crops. However, cassava is not adequately covered within pesticide residue monitoring programmes. This negligence poses a risk to the food safety policy, consumer protection and environmental management, particularly in regions with extensive use of pesticides. This study will focus on assessing pesticide residue levels and the types of pesticides in soil and cassava tubers in a chosen agricultural field.

Methods

Study area

This study was conducted in Obiaruku which is located in the Ukwuani Local Government Area of Delta State, Nigeria. The area is noted for agricultural activities in southern Nigeria, and the flat terrain in the area makes this possible. The soil contains high amounts of nutrients coupled with natural drainage systems which prevent waterlogging and promote the distribution of nutrients in the farmlands (Wizor and Okugin, 2020).

Sample collection and preparation

A systematic grid-based method was applied to collect soil samples within agricultural fields in Obiaruku, Ukwuani LGA, Delta State, Nigeria to make a representative coverage. On every point, the soil cores were hand cut down to the top of the 15 cm mark with a stainless-steel corer and about 500 g of soil was collected. Samples were put in clean, labelled plastic bags and carried to the lab in a cooler at 4 ˚C. The samples of soil were air-dried in the lab at room temperature. Obvious debris and stones were cut off, and then samples were crushed and sifted back through a 2 mm mesh to provide uniform consistency. The samples of processed soil were stored in clean, airtight containers at -20 ˚C during analysis.

The study area had farm plantations where cassava tubers were harvested. A random selection of tubers was made at every place, and special care was taken not to physically damage them.

The samples were put in labelled plastic bags and transported to the laboratory in a cooler at 4 ˚C. At the lab, the tubers were washed with running tap water to wash off soil and debris, peeled and sliced into smaller pieces using a clean stainless-steel knife. A high-speed blender was employed in homogenising the pieces to get a uniform sample. The cassava was homogenised and kept in airtight containers at -20 ˚C until analysis.

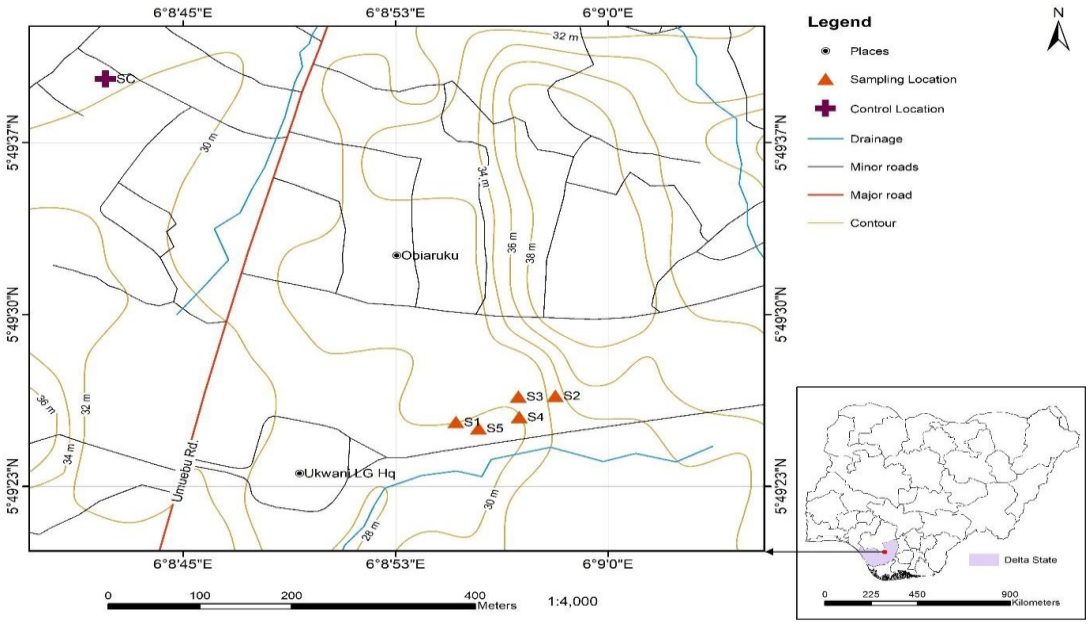


Figure 1: Map of the study area showing sampling locations

Sample size, sample design and analytical procedures

Sampling for the research was carried out at five stations (stations 1 to 5) on agricultural fields at Obiaruku, Ukwuani LGA, Delta State, Nigeria. In addition, cassava tubers were collected at the same farms where the harvesting was done. For each of the five stations, soil samples were taken up to 15 cm depth with a stainless-steel core and transferred to clean, labelled polythene bags while transporting them to the laboratory at 4 °C. Similarly, random selection of cassava roots was made and transported to the laboratory after washing and peeling off the outer layer. Soil samples collected in the laboratory were allowed to dry in ambient air, debris was removed, and sieving was done using a 2 mm sieve to ensure consistency in size. Prepared samples were kept frozen at −20 °C until analysis. The determination of pesticide content was done using GC–MS analysis. Comparison of mass spectra with reference standards was used to identify the compounds present. Quality control procedures adopted involved the use of calibration standards, blanks, and spiked samples.

Analysis of Pesticides

Sample extraction

Pesticides in soil samples were extracted using acetone as the solvent. Soxhlet extraction was

done so as to have complete extraction of the soil matrix. To purify the solvent and make the sample ready to analysis, the resulting extract was rotary evaporated to remove the solvent. In the case of cassava tubers, the extraction solvent was methanol. The extraction process was further enhanced using ultrasonication to maximise the extraction of the pesticide residues.

Pesticide quantification

The target pesticides were quantified by Gas Chromatography-Mass Spectrometry (GC-MS) (Wong et al., 2011; Liu et al., 2020). The GC-MS system was equipped with a capillary column (30 m×0.25 mm, 0.25 mm of film thickness) and worked as follows: injector temperature set at 250˚C, oven temperature programmed (initially at 80˚C and maintained for 2 minutes), ramped at 10˚C /min to 300 ˚C, and maintained at 300 ˚C. The carrier gas was helium with a steady flow of 1 mL/min.

Each pesticide was converted into a calibration standard, which determined the linear range of the method and sensitivity. Sample results were compared with the reference standards to identify retention times and mass spectra of the analyte. The quantification was done using peak areas, whereby the calibration curves were determined using the standards. The concentrations of pesticides in the soil and cassava samples were calculated using the linear regression equations of these curves. The GC-MS system was used in electron ionisation (EI) mode with an ion source temperature of 230 ˚C and a transfer line temperature that was set to 280˚C. The mode was selected as snow ion monitoring (SIM) mode. The validation of methods involved the use of limits of detection (LODs) of each pesticide, which were calculated on a 3:1 signal-to-noise basis. Depending on the compound used, the LODs of pesticide residues were 0.01 to 0.05 mg/kg, which made it possible to detect pesticide residues in soils and cassava samples that contained traces of pesticides.

Quality assurance and control

Calibration of the GC-MS instrument was performed by using standard pesticide solutions spanning the anticipated concentration range in samples. Method validation was done by spiking samples that had known concentrations of pesticides to check the accurate quantification and recovery. The quality control was strictly followed to retain the accuracy and precision of the analysis. Maps were used as the blank and control use of the samples to trace possible contamination or interference. Reproduction tests were done to determine the reproducibility of the results.

Data analysis

Statistical analysis of all data that were taken in the laboratory tests was carried out. Descriptive (mean and SD), as well as inferential statistics (ANOVA, factor analysis and principal component analysis), were performed.

Results

Pesticide residues in soil

Figure 2 shows data of the different pesticides present in the soil of agricultural fields in Obiaruku, Ukwuani LGA, Delta State, Nigeria. This pesticide 1,3-dimethyl-2-Nitrobenzene was observed at all stations except the control with the highest mean concentration recorded at Station 2. In all the stations, including the control, diazinone and Isazophos were not observed. Chloropyriphos-methyl was also found at Stations 1 to 3, not at Stations 4 and 5, and in the control. Pirimiphos Methyl was present in Stations 1, 3 and 4 with the highest concentration in Station 4. Only stations 1 and 4 were positive with station 1 containing a significantly higher concentration of Fenitrothion. The stations with Pirimiphos Ethyl were 1 and 5, and the concentration in the former was higher.

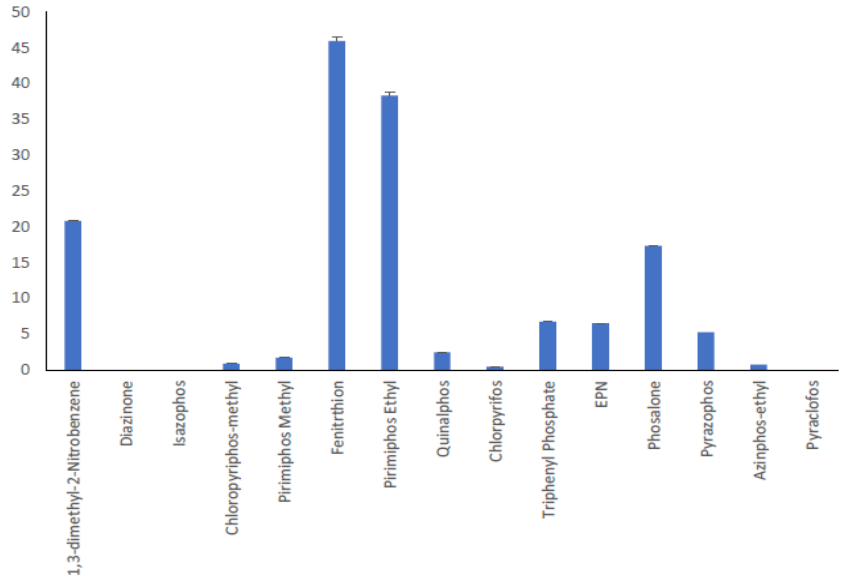


Figure 2: Pesticide residues in soil

Pesticide residues in cassava tubers

The result (Figure 3) indicated that some excellent results existed regarding the pesticide concentrations in cassava crops found in farmlands in Obiaruku, Ukwuani LGA, Delta State, Nigeria. Only in Station 1 and in Station 4 were the 1, 3-dimethyl-2-Nitrobenzene pesticide detected in large amounts and in small amounts in other stations and in the control, respectively. It presents the risk of utilising or contaminating this pesticide in the area. Secondly, concentration was higher in station 1 with moderate levels in station 4, but none in the other stations and the control. This result was similar to that of 1,3-dimethyl-2-Nitrobenzene that revealed that the usage patterns and contamination patterns could be associated. Thirdly, the Pirimiphos Ethyl was observed in high concentration in Station 1 and moderate concentration in Station 5, and not detectable at other stations and the control. This could indicate a different usage trend or a contamination source of this pesticide. Lastly,

Phosalone had only been found in Station 1 and Station 2, with Station 2 having a greater concentration, meaning that its usage or contamination patterns were not the same as those of the rest of the pesticides.

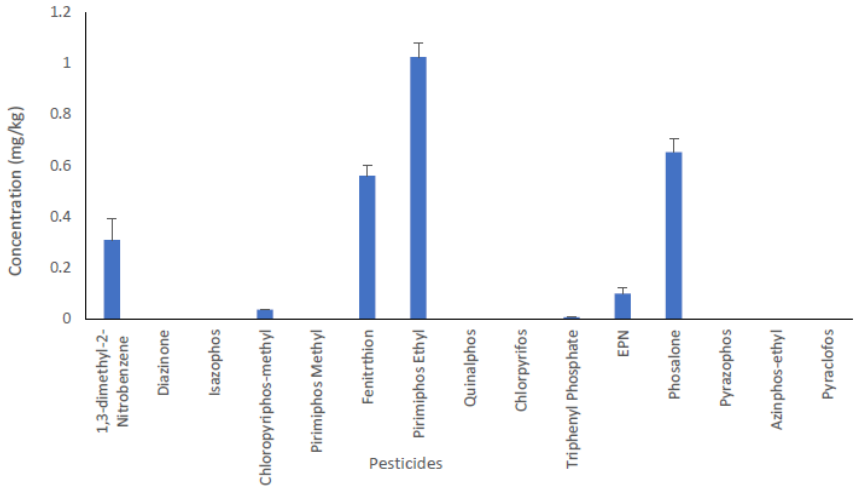


Figure 3: Pesticide residues in cassava tubers

Comparison of pesticide concentrations in soil and cassava tubers

The concentration of different pesticides in the soil and tubers of cassava crops in the agricultural fields within Obiaruku, Ukwuani LGA, Delta State, Nigeria is given in Table 1 below. Mean concentration of the pesticide 1,3-dimethyl-2-Nitrobenzene was 20.85±7.03 in the soil, and it was much less in the tuber (0.31±0.57) ($p<0.05$). This implies that cassava plants may not readily absorb this pesticide from the soil.

The soil had mean concentrations of 45.99 ± 76.62 and 38.32 ± 52.91 of Fenitrothion and Pirimiphos Ethyl, respectively. Nevertheless, they were significantly lower in concentration in the tuber, with a concentration of 0.56 ± 0.80 of Fenitrothion and 1.024 ± 1.65 of Pirimiphos Ethyl($P>0.05$), indicating that these pesticides are absorbed by the cassava plant at a relatively low rate in comparison with the soil.

EPN and Phosalone were found in the soil at moderate mean concentrations of 6.49±3.85 and 17.32±12.05, respectively, and in the tuber at lower concentrations of 0.10±0.22 and 0.65±0.91, respectively ($P<0.05$ for both). This suggests that these pesticides were partially absorbed from the soil by the cassava plant.

Table 1: Comparison of pesticides in soil and cassava tubers

Pesticides	Soil (mean±SD)	Tuber (mean±SD)
1,3-dimethyl-2-Nitrobenzene	20.85±7.03	0.31±0.57
Diazinone	0±0	0±0
Isazophos	0±0	0±0
Chloropyriphos-methyl	0.87±1.03	0.04±0.08
Pirimiphos Methyl	1.71±1.98	0±0

Pesticides	Soil (mean±SD)	Tuber (mean±SD)
Fenitrthion	45.99±76.62	0.56±0.80
Pirimiphos Ethyl	38.32±52.91	1.024±1.65
Quinalphos	2.47±3.19	0±0
Chlorpyrifos	0.45±1.01	0±0
Triphenyl Phosphate	6.75±7.12	0±0
EPN	6.49±3.85	0.10±0.22
Phosalone	17.32±12.05	0.65±0.91
Pyrazophos	5.29±6.88	0±0

Percentage composition of pesticides in samples

The results in Figure 4 show the average percentage composition of various pesticide residues in the soil of cassava crops in Obiaruku, Ukwuani LGA, Delta State, Nigeria. The phenitrothion is the most prevalent pesticide at more than 40 per cent. This high degree is an indication of excessive application of fenitrothion in the area of farming. Other pesticides such as Pirimiphos Methyl and chlorpyriphos-methyl have high quantities as well and therefore, signify high usage of pesticides in this region. However, pesticides such as pyraclofos, azinphos-ethyl and pyrazophos were present in moderately low concentrations.

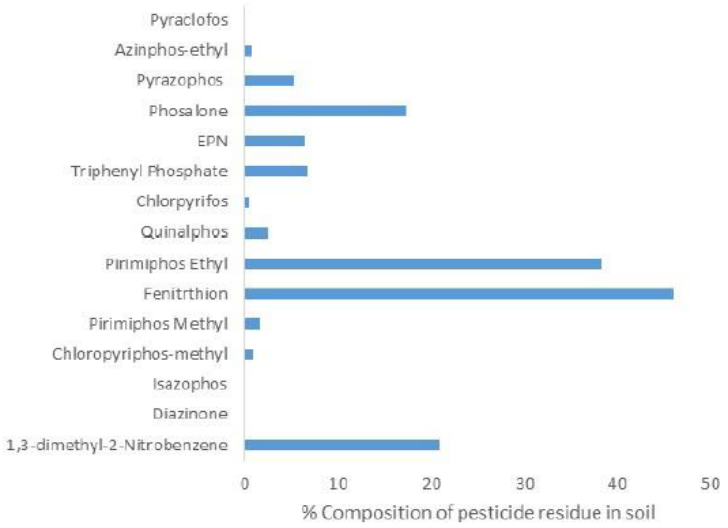


Figure 4: Percentage composition of pesticide residues in soil

The mean percentage composition of the different pesticide residues in the cassava crop tubers is given in Figure 5. The most common pesticides (more than 0.8 percent) were Pirimiphos Methyl and Fenitrothion. This implied that the tubers contained a lot of these pesticides. Large amounts of circa 0.6 per cent also appear with chlorpyrifos-methyl and 1, 3-dimethyl-2-nitrobenzene. However, other pesticides such as Pyraclofos, Azinphos-ethyl, Pyrazophos, Phosa lone, EPN, Triphenyl phosphate, Chlorpyrifos, Quinalphos Ethyl, Pirimiphos Ethyl, Isazophos, diazinone have much lower concentration (less than 0.4 percent). The specified

outcomes represent the general prevalence of certain pesticides and the potential impact that microparticles of this kind have on the quality of the cassava crops.

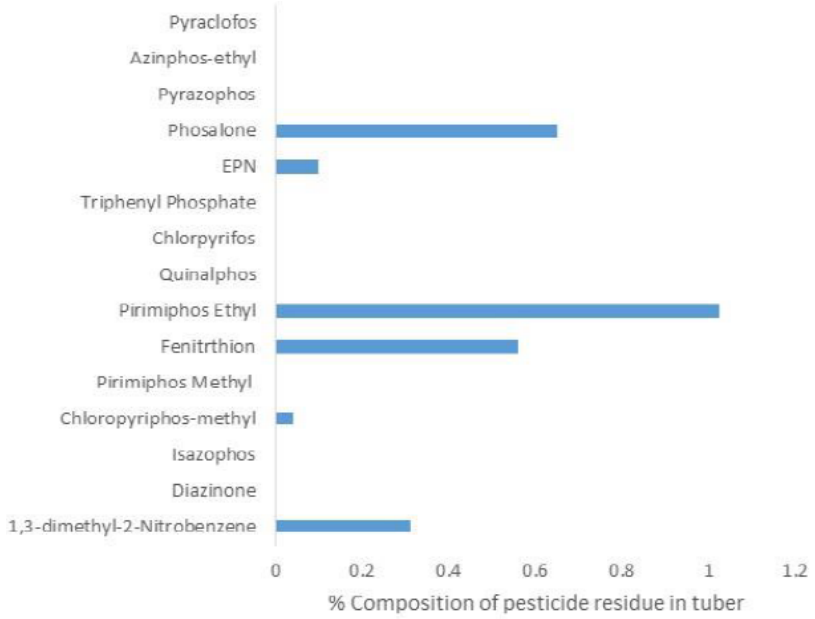


Figure 5: Percentage composition of pesticide residues in cassava tubers

PCA of pesticides in samples

The Principal Component Analysis (PCA) and Factor analysis are presented in Table 2 and provide an overall understanding of how various pesticide residues are distributed in the soil and the relationship between them. The first three components have demonstrated that the data variance was highly contributed by the PCA. Surprisingly, the first constituent had remarkably high levels of pesticides such as Chlorpyrifos, Fenitrothion, Pyrazophos, and Azinphos-ethyl, suggesting a high level of correlation. Quinalphos and 1,3- dimethyl -2- Nitrobenzene had high loadings in the second component, whereas Pirimiphos Methyl had high loading in the third component. Factor Analysis gave different scores of factors within different soil stations. To illustrate, Station 1 was rated high in Factor 1 and Factor 3 and negative in Factor 2. Factor 2 and Factor 3, on the contrary, were high and low in Station 2, respectively. Such differences suggest different distributions of pesticide residues on the stations.

Table 2: PCA of pesticides in soil

	Component		
	1	2	3
1,3-dimethyl-2-Nitrobenzene	.421	.900	.112
Chloropyriphos-methyl	.880	.337	-.300
Pirimiphos Methyl	.569	.152	.724
Fenitrthion	.933	.084	.347

		Component	
Pirimiphos Ethyl	.628	-.522	.099
Quinalphos	-.480	.837	.260
Chlorpyrifos	.983	.019	.076
Triphenyl Phosphate	-.647	-.289	.572
EPN	.917	-.192	-.085
Phosalone	-.396	.832	-.143
Pyrazophos	.956	.179	-.225
Azinphos-ethyl	.983	.019	.076
Extraction Method: Principal Component Analysis.			
a. 3 components extracted.			

Principal Component Analysis (PCA) and Factor Analysis, illustrated in Table 3, indicate the distribution and correlation of various pesticide residues in the tuber. The PCA one and two factors have indicated that they have a significant percentage of variance in the data. It should be mentioned that the 1,3-dimethyl-2-Nitrobenzene, the Fenitrothion, Pirimiphos Ethyl and the EPN and Phosalone reported a significant correlation on the first component. The second component was loaded with a high concentration of Phosalone and Chloropyriphos-methyl. Factor Analysis of the different tuber stations showed different factors. To illustrate, Station 1 has high marks on Factor 1 and low marks on Factor 2. On the other hand, Station 2 had a high score on Factor 2 and a low score on Factor 1. Such differences suggest different distributions of pesticide residues on the stations.

Table 3: PCA for pesticide residues in cassava tubers

	Component	
	1	2
1,3-dimethyl-2-Nitrobenzene	.997	.025
Chloropyriphos-methyl	-.328	.941
Fenitrthion	.881	-.149
Pirimiphos Ethyl	.930	-.026
EPN	.985	.091
Phosalone	.378	.926
Extraction Method: Principal Component Analysis.		
a. 2 components extracted.		

Discussion

Pesticide residues in soil

Soil samples at different sites yielded a response of diverse levels of pesticides on the ground (González-Curbelo et al., 2022). The uniform finding of 1,3-dimethyl-2-nitrobenzane in all places except the control is an indication of extensive use or contamination (George Fouad, 2022). Diazinon and Isazophos may not have been used, or they may have been quickly degraded. The preferential appearance of pesticides like Chlorpyrifos-methyl, Pirimiphos-

methyl and Fenitrothion is indicative of varying use patterns or environmental longevity. These differences are caused by application practises, degradation rates, and environmental factors, which represent the necessity of periodic monitoring and risk assessment (Bernardes et al., 2015).

Nigerian soils contain organophosphate residues, whereas the health and environmental hazards are known (Awe et al., 2022; Fatunsin et al., 2020; Chewe et al., 2019; Mahmud et al., 2015; Mazlan et al., 2017). Certain results indicate concentrations above Maximum Residue Limits (MRLs) which pose the risk of safety concern. As an example, the level of pesticides in soil and crops exceeded MRL in Araromi farm settlement, Osun State and Gashua, Yobe State (Awe et al., 2022; Mahmud et al., 2015). The residues can present occupational health risks of exposure to workers of the farms, as well as to local communities by dermal contact, inhalation, or ingestion (Olowogbon et al., 2021). Ecological risks also play an important role, such as pesticides such as chlorpyrifos, which harbour non-target species such as birds and fish, as well as beneficial insects (Brhich et al., 2022).

Pesticide residues in cassava tubers

Analysis of cassava tubers from farms in Obiaruku, Ukwuani LGA, Delta State, revealed notable pesticide residues, including 1,3-dimethyl-2-nitrobenzene, Fenitrothion, Pirimiphos-ethyl, and Phosalone. Their distribution was localised, implying that they have been locally used or contaminated (Omeje et al., 2024). The varying concentrations of organophosphate (OP) reduction of crops are determined by variations in agricultural activities, insect stress, and the type of crops (Raut, 2020). The degradation of pesticides due to environmental factors like the type of soil used and climate also affects persistence and crop contamination (Mulla et al., 2020).

The extensive application of pesticides in agriculture has caused contamination of soil, affecting the ecosystems and human health. Research indicates that ongoing pesticide residues lower soil quality (Beaumelle et al., 2023; Froger et al., 2023). High levels of 1,3-dimethyl-2-nitrobenzene and Fenitrothion may impair the health of the soil and influence microflora (Walder et al., 2022; Mahdavi et al., 2024). The possibility of health danger in the case of ingesting cassava with high pesticide levels is the element of health risk, such as cancer and neurological issues (Narayanan et al., 2022).

Comparison of concentrations of pesticides in samples

The research at Obiaruku, Ukwuani LGA, Delta State, Nigeria, found significant levels of pesticides such as 1,3-dimethyl-2-nitrobenzene, Fenitrothion, Pirimiphos-ethyl, EPN and Phosalone in the soil and cassava tubers. The results also indicate that cassava plants take up pesticides from the soil, albeit not in a one-to-one ratio.

In previous studies, the presence of such pesticides as imidacloprid, avermectin, carbendazim, glyphosate, thiamethoxam, and lambda-cyhalothrin in soils and agricultural products has been reported (Danek et al., 2020; Jing et al, 2021; Tang and Maggi, 2021). It is also mentioned in

global assessments that there is extensive soil pollution by such compounds as pendimethalin, glyphosate, paraquat, chlorpyrifos, and chlorothalonil in different types of cropping (Jing et al., 2021). These results represent the complex relationships between certain pesticides and crops in which uptake and accumulation are different depending on the compound.

The study also determined that, in cassava tubers, there are lower pesticide concentrations than those in the surrounding soil, which suggests that little is transferred to the portions of the food (Rosiji et al., 2018). Although this can minimise direct human exposure (Wahengbam et al., 2023), soil contamination remains a threat to ecology, such as the loss of biodiversity and soil degradation (Abbou et al., 2023). Such findings are important to understand the dynamics of pesticide uptake and adopt sound environmental and health practises to minimise the risks to the environment and humans.

PCA of pesticides in samples

Principal Component Analysis (PCA) and Factor Analysis provided important patterns of distribution and interaction of the pesticide residues in the soil and cassava tubers of farms at Obiaruku, Ukwuani LGA, Delta State, Nigeria. Chlorpyrifos, Fenitrothion, Pyrazophos, and Azinphos-ethyl are detected in soil samples, which suggests a high level of contamination and environmental distribution (Mahdavi et al., 2021; Sujeeth et al., 2023). The correlations and high loadings of pesticides 1,3-dimethyl-2-nitrobenzene and Quinalphos indicated extensive occurrences in the soil (Bejger et al., 2021; Mahdavi et al., 2021). This indicates the presence of their availability and persistence in the environment (Bhende et al., 2022). The accumulation is affected by such factors as sorption-desorption behaviour, properties of soils, and chemistry of pesticides (Zhichkina et al., 2020). These results highlight the necessity of frequent surveillance to assess the risks of contamination and impose safety precautions. High levels of 1,3-dimethyl-2- nitro benzene, Fenitrothion, Pirimiphos-ethyl, EPN and Phosalone on the first component suggest that they have a strong correlation in cassava tubers (Sujeeth et al., 2023). This indicates that they can be found in edible components, which could have an impact on crop quality and safety (Houngue et al., 2018).

Conclusion

This study aimed at the analysis of pesticide residues in the soil and cassava roots of Obiaruku farms in the Ukwuani LGA, Delta State, Nigeria, to determine the magnitude of contamination and effects on the environment and human health. The results of the research indicated that large amounts of pesticides in soil and cassava revealed strong associations with such pesticides as 1,3-dimethyl- 2-Nitrobenzene, Fenitrothion, and Pirimiphos-ethyl. Although transfer of pesticides to edible parts of cassava was minimal, soil contamination is unsafe to food security and the ecosystem. Due to the long-term presence of some pesticides in the environment, the use of sustainable farming methods must be ensured. Close supervision, appropriate utilisation of pesticides and education of the people must be accorded first priority to curb the dangers. Rates of degradation of pesticides and safe substitutes to pesticides in the

control of pests should also be a research topic.

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